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Inoue & Associates

NO. 2105 P. 14 — — —
NO. 0168 P. 2/7

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of Matsukawa, et al. Docket No: 21581-00314-US

Serial No.: 10/743,387 Art Unit: 1742

Filed : December 23, 2003

Examiner: Daniel J. Jenkins

Title : PRETREATMENT METHOD FOR COATING

DECLARATION UNDER RULE 132

Honorable Commissioner of Patents and Trademarks,
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Futsuhara Masanobu, a citizen of Japan and having postal mailing address of 4-202, Miho-cho, Midori-ku, Yokohama, 226-0015, JAPAN, declare and say that:

In March 1997, I was graduated from Nagoya University;

I am one of the inventors of the above-identified application and am familiar with the subject matter thereof;

I have read the Official Action mailed and the references cited therein and I am familiar with the subject matter thereof;

I respectfully submit herewith my exact report thereon;

The following experiments were conducted by me or under my direct supervision.

(Test for cold-rolled steel, CRS)

A commercially available cold-rolled steel sheet (SPCC-SD, manufactured by Nippon Testpanel Co., Ltd., 70 mm × 150 mm × 0.8 mm) was used as a material, and pretreatment of coating was applied to the material in the following conditions.

(1) Pretreatment of Coating

Degreasing treatment: The metal material was immersed at 40 °C for 2 minutes with 2% by mass "SURF CLEANER 53" (degreasing agent manufactured by Nippon Paint Co., Ltd.).

Rinsing with water after degreasing: The metal material was rinsed for 30 seconds with a spray of running water.

Chemical conversion treatment: A chemical conversion coating agent, having the zirconium concentration and the amino group-containing silane coupling agent concentration shown in the table 1, was prepared by using fluorozirconic acid and

aminoethylaminopropyltrimethoxysilane (KBM603) as the amino group-containing silane coupling agent. A pH was adjusted to be 4 by using sodium hydroxide. The temperature of the chemical conversion coating agent was controlled at 40 °C and the metal material was immersed for 60 seconds.

Table 1

Test No.	Concentration of components (ppm)		
	H ₂ ZrF ₆	Zr	KBM603
1	1000	440	2000
2	3000	1319	5000
3	5000	2198	10000
4	1137	500	250
5	500	220	250

Rinsing after chemical conversion treatment: The metal material was rinsed for 30 seconds with a spray of running water. Further, the metal material was rinsed for 10 seconds with a spray of ion-exchanged water. Then, electrocoating was applied to the metal material in a wet condition. It is noted that a coat amount was analyzed as an amount of zirconium contained in the chemical conversion coating by using "XRF-1700" (X-ray fluorescence spectrometer manufactured by Shimadzu Co., Ltd.) after the cold-rolled steel sheet after rinsing was dried at 80 °C for 5 minutes in an electrical dryer.

(2) Coating

Electrocoating was applied to the surface in such a manner that a dried film thickness was 20 µm using "POWERNIX 110" (a cationic electrodeposition coating composition manufactured by Nippon Paint Co., Ltd.) and, after rinsing with water, the metal materials were heated and baked at 170 °C for 20 minutes and test sheets were prepared.

Evaluation Test

<Secondary adhesion test (SDT)>

Two parallel lines, which have depth reaching the material, were cut in a longitudinal direction on the obtained test sheet and then the test sheet was immersed at 50 °C for 480 hours in 5% aqueous solution of NaCl. After immersion, a cut portion was peeled off with an adhesive tape and peeling of a coating was observed.

◎: No peeled

○: Slightly peeled

X: Peeled 3 mm or more in width

Results of observations are shown in Table 2.

(Test for galvanized steel sheet, GA)

The test sheet was prepared by following the same procedure as that of (Test 1-1) except that the metal material was changed to galvanized steel sheet (GA steel sheet, manufactured by Nippon Testpanel Co., Ltd., 70 mm × 150 mm × 0.8 mm), and evaluation was carried out.

(Test for aluminum)

The test sheet was prepared by following the same procedure as that of (Test 1-1) except that the metal material was changed to 5000 series aluminum (manufactured by Nippon Testpanel Co., Ltd., 70 mm × 150 mm × 0.8 mm), and evaluation was carried out.

Table 2

Test No.	Coat amount (mg/m ²)			SDT			SST		
	CRS	GA	Al	CRS	GA	Al	CRS	GA	Al
1	10.7	4.00	9.00	×	○	○	×	○	○
2	3.16	2.30	3.40	×	○	○	×	○	○
3	1.50	1.98	2.70	×	○	○	×	○	○
4	72.1	42.7	43.0	◎	◎	◎	◎	◎	◎
5	22.0	14.9	11.9	◎	◎	◎	◎	◎	◎

(Results)

From Table 2, even if a chemical conversion coating agent containing too much amount (identical amount as WO'399) of amino silane from the amount in claim 1 (Test No.1 to 3) is applied to aluminum material, preferable results are obtained.

However, when such a chemical conversion coating agent is applied to iron material, preferable results are not obtained in SDT test and SST test.

When the concentration of amino silane is adjusted to concentration of the present invention (Test No.4 and 5), the test results are absolutely different. Good results are obtained for all of these three substances. Further, for GA and aluminum, the test results of SDT and SST are improved.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and

further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed this 15 day of March, 2006

Futsuhara Masanobu

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